Piezoelectric Actuation of Graphene-Coated Polar Structures

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***Abstract*— Ferroelectric materials based on lead zirconate titanate (PZT) are widely used as sensors and actuators because of their strong piezoelectric activity. However, their application is limited because of the high processing temperature, brittleness, lack of conformal deposition, and a limited possibility to be integrated with the microelectromechanical systems (MEMS). Recent studies on the piezoelectricity in the 2-D materials have demonstrated their potential in these applications, essentially due to their flexibility and integrability with the MEMS. In this work, we deposited a few layer graphene (FLG) on the amorphous oxidized Si_3N_4 membranes and studied their piezoelectric response by sensitive laser interferometry and rigorous finite-element modeling (FEM) analysis. Modal analysis by FEM and comparison with the experimental results show that the driving force for the piezoelectric-like response can be a polar interface layer formed between the residual oxygen in Si_3N_4 and the FLG. The response was about 14 nm/V at resonance and could be further enhanced by adjusting the geometry of the device. These phenomena are fully consistent with the earlier piezoresponse force microscopy (PFM) observations of the piezoelectricity of the graphene on SiO_2 and open up an avenue for using graphene-coated structures in the MEMS.**

***Index Terms*— Finite-element analysis, graphene, membrane, piezoelectricity, resonance.**

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I. Introduction

CURRENTLY, inorganic ferroelectrics such as lead zirconate titanate (PZT), lithium niobate (LN), and barium titanate (BT) are widely used as efficient piezoelectrics, pyroelectrics, memory cells, and electrooptic modulators [1]. They possess high switchable polarization, strong piezoelectric response, and remarkable pyroelectric and electrooptic properties, but have a number of disadvantages that preclude their use [2], [3]. First, they are not biologically compatible and require encapsulation for contact with biological environments. Second, their processing is at high temperatures, so their miniaturization and integration with the microelectromechanical systems (MEMS) are difficult. Recent discovery of piezoelectricity in 2-D materials [4], [5] opens up new opportunities for stretchable electronics [6], MEMS, and other electronic components based on the direct and converse piezoelectric effects. Being a 2-D monoatomic material with many unique properties [7], graphene is one of the favorable candidates for these applications. It exhibits a variety of emergent properties such as high thermal conductivity, superior mechanical strength, and extremely high flexibility. Although pristine graphene does not possess any piezoelectric activity due to its intrinsically centrosymmetric crystal structure, polar properties (including piezoelectricity) can be induced by breaking of the inversion symmetry by the adsorption of foreign atoms, by introducing specific in-plane defects, or by nonuniform deformation of graphene layers, in which strain gradients create internal polarization in a material [8]–[11]. Recent studies [12] revealed a strong piezoelectric activity of the graphene flakes deposited on the Si/SiO_2 grating substrates. The piezoelectric properties were evaluated by piezoresponse force microscopy (PFM), which is based on the detection of local deformation caused by the small ac bias applied to the conducting tip of the PFM cantilever. The piezoelectric activity in the

graphene layers was attributed to the chemical interaction of the graphene atoms with the underlying oxygen from the SiO_2 substrate [12]. The local piezoelectric coefficient was sufficiently high, comparable with the values for the best piezoelectric ceramics, e.g., PZT or $BaTiO_3$. However, no macroscopic observation of piezoelectricity has been reported until now due to the extremely thin polar layer preventing the measurements of both direct and converse piezoelectric effects.

In this work, we study piezoelectric resonances in thin silicon nitride membranes covered by graphene flakes with the bottom Al electrode. The piezoelectric response was about 14 nm at 3.7 kHz under 1 V applied between the graphene and the 100-nm Al layer. These results provide clear prospects for using graphene as an MEMS material; however, membrane parameters have to be further optimized in order to increase the useful displacements and the desired frequency range.

II. Experiment

In this work, low stress (nonstoichiometric) 1-μm-thick silicon nitride films were deposited onto the Si(100) wafers by means of low-pressure chemical vapor deposition (LPCVD) from a gas mixture of dichlorosilane (DCS), SiH_2Cl_2, and ammonia in a Tystar horizontal furnace. Etch windows were photolithographically delineated on the wafer backsides followed by a plasma etch to remove the Si_3N_4 layer. After that, the Si_3N_4 membranes were manufactured by wet etching of the nonprotected by nitride silicon in hot 30% KOH. The membrane dimensions were 6.7 $\times$ 6.7 mm^2, and the entire surface of the etched area was covered with graphene flakes, as shown schematically in Fig. 1. Several samples were studied by XRD, but no diffraction peaks other than those belonging to the Si substrate were found, indicating that the silicon nitride films were amorphous; the Si crystallites mentioned in literature [13] were not observed. Al electrodes of 100 nm in thickness were sputtered through a mask on the nonetched side of the wafer (Fig. 1). Energy-dispersive analysis of X-rays (EDAX) was used to determine the oxygen concentration of about 0.72 at.% in silicon nitride. Graphene was synthesized on a PlanarTech G2 installation using the CVD method on a copper foil. Acetylene was used as a prosecutor; hydrogen was added to the reactor for dilution in the ratio C_2H_2: H_2 = 1: 4. The process temperature was 1040 °C and the pressure was 6 Torr. The transfer was made using a film of polymethyl-methacrylate (PMMA), as an intermediate substrate. First, a liquid polymer (4% solution of PMMA in anisole) was applied to a copper foil with graphene by centrifugation at a speed of 1500 rpm and was baked in an oven at a temperature of 150 °C. Furthermore, copper was etched away in an aqueous solution of chlorinated iron. Then, the obtained PMMA film was washed twice in deionized water and laid on a substrate. After drying in a centrifuge at a speed of 3000 rpm, the film was heated on a hot plate at a temperature of 120 °C to eliminate the wrinkles. After that, the PMMA layer was removed in acetone. The quality of graphene was checked by Raman scattering (Fig. S1), and it was shown that it is indeed FLG. The measurements of electric field displacements were done using the sensitive

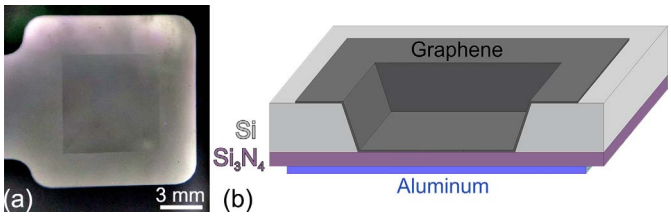


Fig. 1. (a) Optical image of the graphene-coated 6.7 $\times$ 6.7 mm^2 Si_3N_4 membrane (bottom view). (b) Schematic of the membrane (side view).

double-beam laser interferometer [14], [15] with the sensitivity of $2{\cdot}10^{-2}$ Å. The beam of the interferometer was positioned in different places of the membrane. Finite-element modeling (FEM) of the membranes was performed using the COMSOL Multiphysics package (version 5.4).

III. Results and Discussion

As the studied silicon nitride structure is amorphous, no piezoelectric response was expected from the graphene-coated membranes, except electrostriction measured at the second harmonic of the applied ac bias. Contrary to these expectations, a strong response was found at the first harmonic, as shown in Fig. 2. The results clearly demonstrate that, opposite to common knowledge, graphene-coated membranes exhibit a strong piezoelectric activity more than 30 times exceeding that of electrostriction. The piezoelectric signal was linear with the applied bias [Fig. 2(b)], while the signal at the second harmonic demonstrated a pronounced quadratic behavior with the field [Fig. 2(d)]. The calculated effective electrostriction coefficient measured in the center of a membrane was about $(M_{11})_{\text{eff}} \sim 4.4{\cdot}10^{-13}$ m^2/V^2. This value is much greater than that observed in a similar compound SiO_2 ($M_{11} \sim 10^{-15}$ m^2/V^2) [16]. A part of this enhancement could be due to the flexure of the membrane that presents a bimorph structure with the graphene/Si_3N_4 interface as an active layer clamped by the underlying Si_3N_4 and Al electrode.

In addition, in contrast to piezoelectric quartz (d_{11} = 2.3 pm/V), the effective piezoelectric coefficient of the graphene-coated membrane is about 1 nm/V at low frequencies and reached 14 nm/V at the fundamental resonance (Fig. S5). This value is comparable with that necessary for the application of membranes as micropumps for drug delivery and other microfluidic devices. For example, poly(vinylidene fluoride-co-trifluoroethylene) (PVDF-TrFE)-based membranes demonstrate the voltage-induced piezoelectric response of about 20 nm/V [17]. The advantage of graphene compared with PVDF-TrFE is the ease of deposition, no necessity to pole by stretching, and much greater flexibility that will facilitate the fabrication of ultrathin silicon-based membranes with large displacements.

Membranes are always in tension and, in a rough approximation, their fundamental resonance frequency (for the case of a thin square membrane clamped at the edges) can be written as [18]

$$f = \frac{\sqrt{2}}{2L}\sqrt{\frac{T}{\rho t}} \tag{1}$$

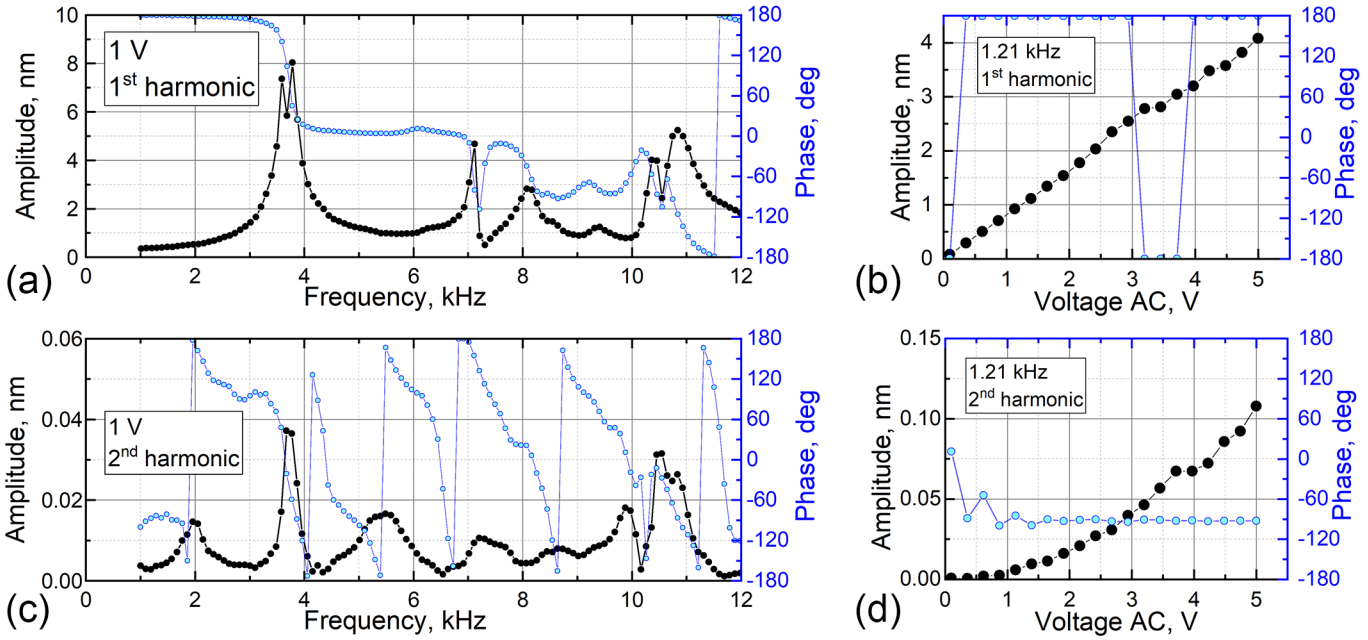


Fig. 2. Frequency dependences of (a) first and (c) second harmonics of the displacement measured in the center of a membrane under an ac voltage of 1 V between the graphene and Al electrodes. AC voltage dependences of the displacements measured under quasi-static conditions at (b) first and (d) second harmonics.

where L is the side length of the membrane, and T, ρ, and t are the tension, density, and thickness of the membrane, respectively. Using this equation, we could estimate the tensile stress to be about 4.7 MPa for our membranes. This sufficiently low stress value could be due to a competition of the compressive stress imposed by the thermal mismatch between the deposited layer and the substrate (introduced on cooling from the deposition temperature) and a larger intrinsic tensile stress, caused by the growth process itself [19], [20]. However, for the rigorous analysis of the piezoelectric displacements induced by graphene, a more quantitative method of FEM is needed as follows.

All the calculations in this work were made by the FEM using a COMSOL Multiphysics software. In the simulation procedure, free (clamped only at the edges) square membrane (with Al electrode) and square membrane with the fixed part (due to the silver-paste contact between the graphene and the contact wire) have been modeled for extracting the natural frequencies and mode shapes during the graphene-excited vibrations (Fig. S4). The dimensions of the membranes have been taken from the experiment. Only two membrane layers, Si_3N_4 layer and aluminum electrode layer, were considered as being significant in membrane mechanics. The contribution from the mechanical properties of the graphene electrode was apparently neglected due to its negligible thickness. To describe the Dirichlet boundary conditions, external edges along the perimeter of the membrane were assumed to be fixed at zero displacement.

For the optimal mesh geometry, a regular quadrilateral prism was used for the elements. The size of the elements does not exceed 150 μm in the plane of the membrane, and at the same time, five elements fit in the membrane thickness (Fig. S2). The initial stress of the Si_3N_4 layer in the membrane was a parameter in the calculations and varied from 0.5 to 6 MPa. The stress was supposed to be distributed uniformly along the membrane plane.

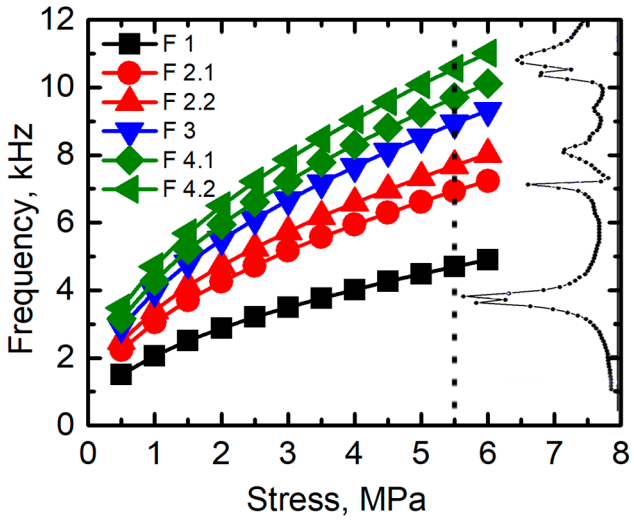
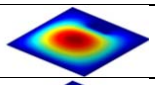
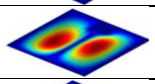
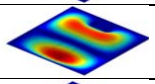
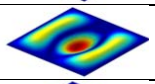
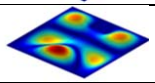
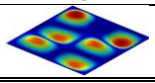


Fig. 3. Calculation results comparing the stress dependence of the piezoelectric resonance of the Si_3N_4 membrane with the clamped electrode (silver paste) with the real resonance spectra. It is seen that the calculated frequencies match the experimental one (right) for the stress of about 5.5 MPa.

The first calculation set was made for the free membrane [Fig. S4(a)]. Comparison of the calculated stress dependences of the natural frequencies with experimental vibration spectrum did not match all the resonant peaks. Taking into account the fixed part 1.2 $\times$ 1.2 mm^2 on the top of the membrane [Fig. S3(a)], which corresponds to the glued electrode used in

TABLE I
COMPARISON OF THE CALCULATED AND EXPERIMENTALLY MEASURED RESONANCE FREQUENCIES OF GRAPHENE-COATED MEMBRANES

Resonance peak	Experimental frequency, kHz	FEM frequency at 5.5 MPa	Type of resonance	Splitting	Amplitude, nm
Fundamental frequency	3.77	4.71		no	13.6
2nd peak	7.11	6.96		yes	7.9
3rd peak	8.08	7.73		yes	2.46
4th peak	9.40	8.98		yes	10.9
5th peak	10.36	9.74		no	4.01
6th peak	10.75	10.6		no	7.5

the experiment, gave a complete coincidence of the number of peaks and the number of the natural frequencies in the studied frequency range (Table I). Increasing the natural frequency number is due to the membrane symmetry breaking, which leads to the splitting of the second vibration mode (Fig. 2) and the appearance of the new modes at higher frequencies. The peak splitting could be caused by two reasons: 1) the intrinsic elastic anisotropy present in Si can result in a distribution in the membrane curvature as a function of in-plane angle even if the overlying Si_3N_4 film is isotropic (amorphous) and 2) asymmetric contact on the graphene [Fig. S3(a)] that results in the asymmetric mode shapes as confirmed by FEM modeling [Table I and Fig. S4(b)]. In order to account for the tensile stress on the membranes, we varied the stress in the FEM calculations (Fig. 3). The best match with the experiments was found for the mechanical stress value 5.5 MPa, which is very close to that calculated with (1). A comparison of the experimental values of resonance frequencies with those obtained by FEM is presented in Table I. Relatively good agreement between the calculated and experimental values is seen (especially for higher modes). This can be explained by the inhomogeneous stress

Fig. S(5) shows the frequency dependences of the piezoelectric resonances measured across the membranes in five points. The measurements were done in order to match the resonance mode shapes [Fig. S4(b)] with the experimental results. Some of the resonance peaks are suppressed and some are amplified. This is because the measurement points (1–5) are located close to the nodes and antinodes of the vibrating membrane. Overall mode shapes shown in Table I are very close to the experimental ones, thus validating the used FEM approach.

As can be noticed from (1), further increase in the resonance frequency of the membranes could be achieved by decreasing their lateral dimensions, while keeping the tensile stress small enough to prevent breaking of the membranes. Optical lithography extended its resolution capability down to 250 nm, and using the ArF excimer laser (193 nm) could further decrease the size of the membranes. As it is well known, X-ray lithography can be used to get the membrane size down to a few tens of nanometer. However, these structures are becoming difficult to fabricate due to the high aspect ratio. As such, the membranes with a thickness of several nanometers and 10 nm in size are currently feasible to combine with the graphene layer. This would extend the resonance frequency to the megahertz range. Control of the membrane stress is also essential in this case [20]. To fabricate thin Si_3N_4 membranes with a thickness of less than 5 nm in a wafer, a new fabrication process that employs a polycrystalline-Si (poly-Si) sacrificial layer has been proposed and evaluated [21]. This fabrication process significantly minimizes damage to the membrane. Using this process, Si_3N_4 membranes with the thicknesses of 3 nm were stably fabricated with small thickness variation.

One of the possible applications of the fabricated membranes is in the area of the DNA sequencing. In the 1990s, it was proposed that it might be possible to use nanopores as sensors for DNA [22]. If DNA could be transferred through a nanometer-sized nanopore in a linear fashion, this might serve as a device to read out the sequence of DNA in an ultrafast way, which is of obvious interest for genomics applications. Current readout of the nanoporous membranes is based on the measurements of ionic conductivity [23], while piezoelectric readout could be much faster and reliable. If sequencing with nanoporous membranes covered by graphene turns out to be feasible, the impact will be huge because it will remove the need for chemical modification, amplification, and surface adsorption of the DNA. Single-molecule DNA sequencing could be one of the promising directions. Further investigations are needed in this field.

On the other hand, membranes with lower resonance frequency might be required for biomedical applications, for example, for vibrational energy harvesting [24]. This could

be achieved by thinning the membranes while increasing their size. However, this could turn their low tensile stress into compressive one and cause buckling [20]. Obviously, more efforts are needed in this direction.

IV. Conclusion

In this article, we prepared piezoelectric membranes consisting of the oxidized amorphous Si_3N_4 membranes sandwiched between a few layer graphene and Al electrodes. Such membranes exhibit clear piezoelectric resonances that were rationalized by the FEM calculations. The highest displacement in the center of a square membrane of 6.7×6.7 mm^2 was about 14 nm/V and could be further enhanced by adjusting the geometry of the device. Such a displacement is comparable with that exhibited by the polymer membranes based on PVDF-TrFE and could be used in flexible/stretchable electronics, for example, in microfluidic devices or electric energy harvesting. Furthermore, such membranes should be sensitive to the mechanical stress and can be used as biocompatible sensors or future devices for DNA sequencing.

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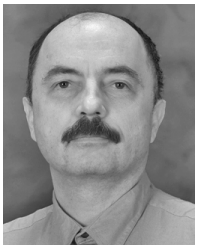

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